

SURFACE HARDENING AND MICROSTRUCTURAL EVOLUTION OF HIGH CARBON STEEL BY FRICTION STIR PROCESSING

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ABSTRACT

In this investigation, effect of single pass FSP with Varying RPM of 150, 300 and 600 at a fixed feed rate of 70 mm min⁻¹ were studied for C70 steel which is majorly used in the automotive industry. Mechanical properties such as micro hardness and tensile strength along with microstructural refinement are investigated in depth by Scanning electron microscope and optical microscope. FSP resulted in three times enhanced micro hardness in comparison to BM with 220 HV hardness. A similar result was achieved with significant increase in the UTS of FSPed sample with 1190 MPa in comparison to a low UTS of 640 MPa of BM. A relative reduction of 50% in elongation of FSPed sample was recorded in comparison to 14% of BM. Increased strength was primarily attributed to phase transformation of ferrites to martensite as a result of intense plastic deformation followed by rapid cooling thereby reducing the grain size from 20µm to 2-3µm in accordance with renowned Hall-Petch equation.

KEYWORDS: High Carbon Steel, Friction Stir Processing, Mechanical Properties, Ultrafine Grain Structure & Microstructure

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INTRODUCTION

Friction stir processing is a surface refinement technique whose basic principle is imitated from friction stir welding. FSW was patented by the welding institute and introduced in 1991[1][2]. This technique of joining metals was found to be very useful as it could join alloys which were difficult to weld using conventional methods. FSP is a solid-state method which is used to create external nanostructure layers on various materials like alloys of aluminum, titanium or different grades of steels. Friction stir processing's main purpose is to ameliorate surface properties by generating surface layers, microstructural refinement, elimination of defects caused by manufacturing processes and reducing grain size. A non-consumable tool held on CNC operated milling machine is penetrated inside the substrate of metal or alloys as shown in Figure 1 in which 1- fixture for holding substrate, 2- C70 Substrate, 3- Advancing side, 4- W-C Tool, 5- clamps for holding the substrate and it is made to rotate on its surface in stirring motion and in transverse direction which therefore, generates heat due to friction and shoulder avoids the plasticized material to flow outwards[3][4][5].

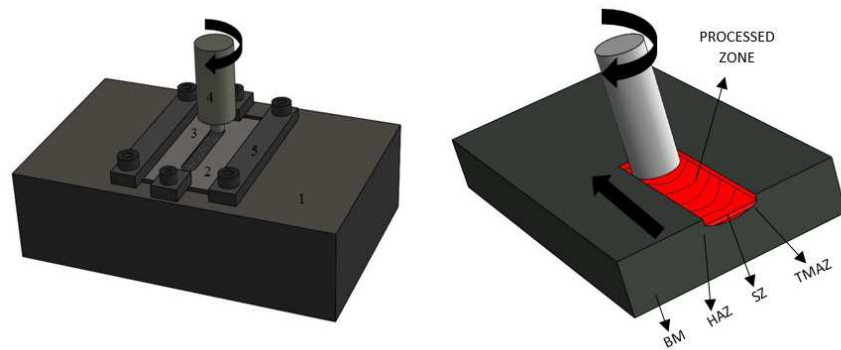


Figure 1: Schematic Illustration of Present Work

FSP of steel tool is designed with a pin or pin less, usually made up of tungsten carbide (WC) or polycrystalline cubic boron nitride (PCBN). The tool used for FSP plays an important role as it is under constant heavy axial load and friction between tool and substrate produces intense heat, keeping in consideration all the factors. Studies on a range of materials confirmed that the quality of FSP and behaviour of material flow mostly depends on substrate's properties as well as on tool's material, shape and dimensions, processing parameters like rotational speed, feed rate, axial force, cooling system, plunge depth and number of passes[6][7][8][9][10]. The friction generated by Friction stir processing play foremost role in enhancing the properties of substrate through intense modification and localized plastic deformation, thereby reducing and refining the grain size of the substrate[11]. C70 is extensively used in the sector of automobile engineering for the fabrication of connecting rods, performance rods, Torsion and stabilizer bars, Railroad rails and bearings of various automobiles and shows potential in the field of shipbuilding industries. The attainment of elevated temperatures plays an important role in the enhancement and modification of a material's properties. It has been observed all around the globe that machines, tools and apparatus that are being involved in various field of thermal and mechanical technology areas have to work under heavy loads and high pressures[12]. Therefore, the machinery is subjected to heavy loads at high temperatures and likely to face common issue of breakdown, failure and corrosion. In automobile industry connecting rods and bearings as well as gas turbines in aerospace industry are exposed to high pressure and temperatures. In the field of petroleum, chemical, and various power plants, corrosion hinder and considerably reduces the efficiency and finally reduces the life of equipment's[13].

Friction Stir Processing is the new and fast developing technique which is nowadays used for subsurface structural modification of different materials, including not only aluminum and magnesium alloys but also steels and other alloys [14]. FSP is done for intermolecular changes and refinement of microstructures in different zones which are affected by heat produced on the metallic components which ensures enhancement in the mechanical properties[15]. According to the literature survey it has been confirmed that FSP is not only an effective technique to attain microstructural refinement but also densifies and homogenises the processed zone. FSP also contributes in the elimination of manufactured defects. FSP has resulted in improved mechanical properties such as micro hardness, ultimate tensile strength, bending strength as well as provides resistance against tribological properties like corrosion and wear[16]. In contrast, ultrafine microstructures with equiaxed recrystallized grains enhances super plasticity behavior[17][18][19]. Earlier efforts were made in investigating the effects of tool pin shape on microstructure characterization and mechanical properties[20][21]. Figure 2 and 3 shows process of FSP and FSPed C70 samples respectively.

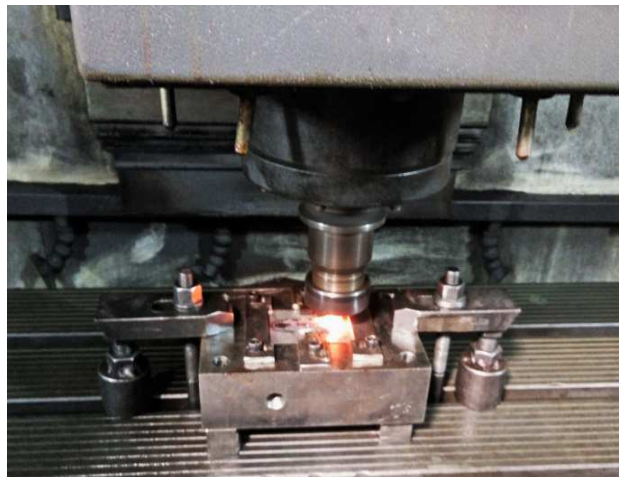


Figure 2: Process of FSP



Figure 3: Friction Stir Processed C70 Specimens

The main objective of the present investigation is to study the effects and correlation of process parameters on the quality of FSP and to enhance the mechanical strength of C70 steel through plastic deformation and dynamic recrystallization and to analyse its effect by optical microscopy (OM) and scanning electron microscopy (SEM), microhardness and tensile testing. The microstructure, mechanical properties of the unprocessed and FSPed materials will be evaluated and compared.

EXPERIMENTAL PROCEDURE

The C70 steel was chosen as substrate material in the current study. The chemical composition of C70 steel is 0.70 wt.%C, 0.80 wt.% Mn, 0.030 wt.%P, 0.045 wt.% C and remaining Fe. The unprocessed substrate C70 steel plates with 100x40x4 mm dimensions were friction stir processed. In the process to minimize the tool wear, selection of tool material, i.e. Pin less Tungsten Carbide of 8mm diameter and 100 mm length was selected with the sole purpose to minimize wear resistance used for working at high temperature. In the current research work the single pass FSP was done at varying parameters as provided in table 1. The fixture for FSP was mounted on VMC 640 CNC operated machine; the tool was

held for a dwell time of 10 seconds to plastically deform the material and was allowed to travel along the slot. After the procedure, specimens were rapidly cooled for 20 minutes at room temperature.

Table 1: Processing Parameters FSP of C70 Steel

Parameters	Value
Tool RPM	150, 300 & 600
Feed Rate	70 mm min ⁻¹
Plunge Depth	1 mm
Axial Pressure	5 KN
No of Passes	1

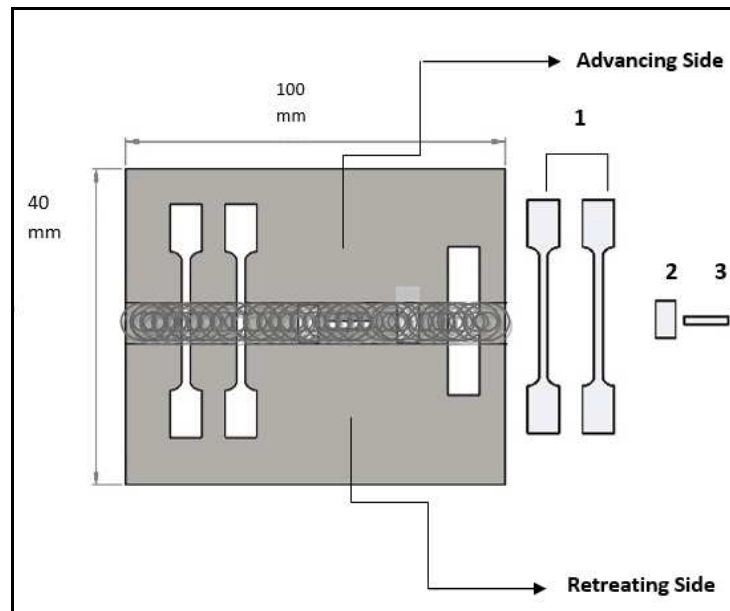


Figure 4: Machining of Samples for Mechanical Testing and Metallographic Analysis

The specimens were cross-sectioned at 90-degree angle to the FSP direction and prepared for metallographies analysis and tensile testing, as shown in Figure 4, hot mounted using mount press machine and polished on SiC emery paper with grades starting from 200 grit followed by 400, 600, 800, 1000, 1200, 1500 and 2000 grit and the final polishing was done with velvet paper to remove scratches prior to which the samples were cleaned ultrasonically with ethanol to remove any dirt or grease. The microstructural characterizations were examined by optical microscopy (OM) on a fully computerized Leica microscope and scanning electron microscopy (SEM) on JEOL JSM-6610LV. Prior to Optical Microscopy the specimens were etched in a solution containing 98% HNO₃ and 2% ethanol. Mechanical characterizations were evaluated using Vickers hardness with a load of 0.3 kgs and tensile testing was done with a strain rate of 1 mm min⁻¹ on fully computerized TINIUS OLSEN H50KS UTM Machine.

RESULTS AND DISCUSSIONS

Tensile Test

Figure 5 shows the tensile stress and strain curves for FSPed and BM and the results are further summarized in table 2. The ultimate tensile strength (UTS) and yield strength (YS) of the C70 steel was approximately 640 MPa and 540MPa and total elongation was approximately 14%. After performing FSP on C70 steel a marginal increase in the YS and

UTS were seen in the samples processed at lower RPM with W-C tool. Whereas, steel processed at higher RPM has shown good results with UTS 1190 and Yield Strength 1080 MPa respectively. However, a sharp decrease in the elongation value from 14% to 7% was seen in higher RPM processed samples. In an investigation by P, Xue et al similar results were seen wherein the tensile ductility of mild steel reduced after FSP with reduced elongation in comparison to BM[22]. In comparison to the base metal of C70 steel the ductility of tensile specimens shows a clear decrement in FSPed zone and high strength was achieved.

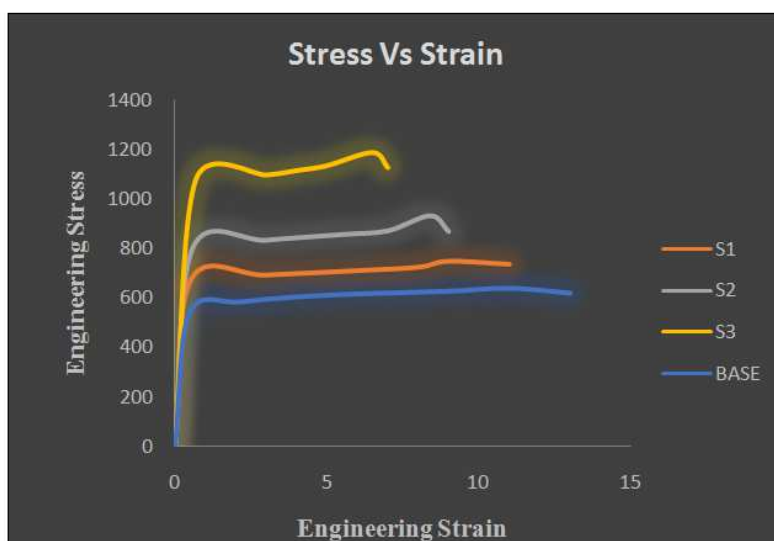


Figure 5: Engineering Stress Vs Engineering Strain Plots for Fsped C70 Steel

Table 2: Tensile and Hardness Values of Fsped Samples

Sample	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)	Hardness _{0.3} (HV)
Base	540	640	14	220
S1	676	750	11	300
S2	805	935	9	406
S3	1080	1190	7	681

From the current experimentation of tensile specimen, elongation was seen to be lower in FSPed region. The results show there is a formation of martensite structure in the processed zone. Figure 6 shows SEM Morphology of the tensile results of FSPed steel which has revealed flat fracture behaviour and no decrement in the area of fractured surface was seen but in the case of BM profound dimples were observed indicating the ductile fracture. The results for FSPed samples shows brittle fracture for C70 steel may also be due to the low elongation and high tensile strength. As discussed above twin phase of ferrite and martensite structure was seen after FSP, it is known that during phase transformation of austenite to martensite more carbon atoms are obtained than ferrite [23].

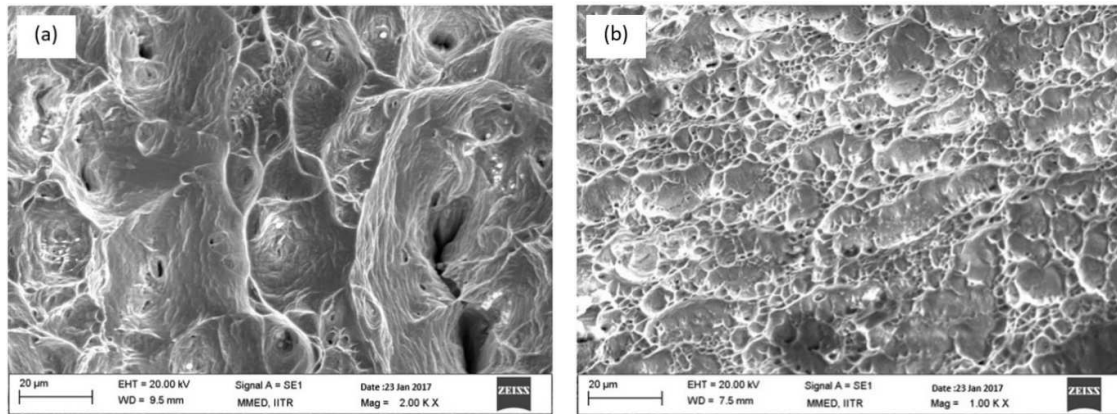


Figure 6: SEM Macrographs of (a) BM with Profound Dimples and (b) Flat Fractured Tensile Specimens

Hardness

The Micro hardness values of BM and FSPed C70 Steel were compared, for the higher RPM processed specimen (S3) the results were found to be 681 HV, 300 and 406 HV for sample S1, and S2 respectively. The observed micro hardness enhancement in the C70 steel may mainly be attributed due to the presence of ultrafine grain structure in the FSPed steel. The hardness profile of samples is shown in Figure 7 and table 3 shows the average and maximum hardness values of processed samples.

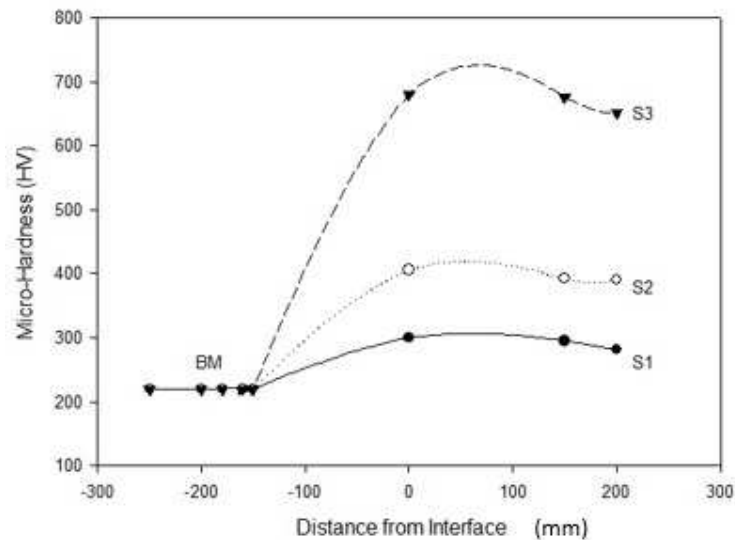


Figure 7: Plots Showing Variation of Hardness along Distance from Interface

Table 3: Average and Maximum Achieved Values of Processed Samples

Sample	Average Hardness (HV)	Maximum Hardness (HV)
Base	197	220
S1	292	300
S2	396	406
S3	669	681

The existence of ultrafine grains for the sample 3 has contributed towards the enhancement of material's strength in accordance with the Hall-Petch Equation as shown in Eq (1),

$$\sigma = \sigma_0 +$$

(1)

Where σ is the yield strength, σ_0 is the stress for dislocation movement also known as frictional stress, k is the strengthening coefficient of material and d is the diameter of average grain size. This equation can be stated for hardness, as in Eq (2),

$$H = H_0 + k_H d^{-\frac{1}{2}} \quad (2)$$

Where H_0 and k_H are constants.

The Hall-Petch equation is usually helpful in determining the relationship between grain size and hardness of the material, as the grain size is reduced there is an increase in the hardness of the material. Experimental values of hardness and observed average grain size of FSPed specimens clearly show the relationship between both in accordance with Hall-Petch Equation.

Figure 8 shows the surface plots describing the influence of Tool RPM on the hardness and grain size of FSPed steel, it represents the simultaneous variation of grain size with respect to RPM and Hardness. Surface plots are helpful in determining the reduced grain size at higher RPM and increased hardness.

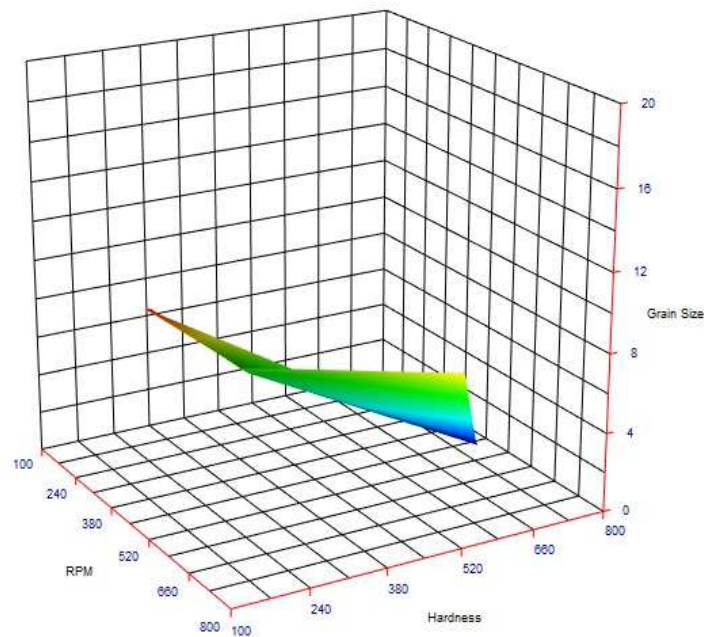


Figure 8: 3-D Plots Showing Influence of RPM on Hardness and Grain Size

The increase in the microhardness value obtained was nearly three times than that of BM. In another investigation, by S. Dodds et al and A.K. Lakshminarayanan et al, a threefold increase in the microhardness was reported at similar parameters [24][25].

Optical Microscopy

The microstructure of C70 steel processed at 600 RPM with grain size ranging between 2-5 μm is shown in Figure

9 in which a clear transition of grains can be seen clearly. The preliminary microstructure of C70 steel contains ferrites with mean grain size of $20\mu\text{m}$ as well as minute grains of pearlite. The microstructure refinement was seen in the processed zone as the grain size was reduced to $4-6\mu\text{m}$ at high RPM. Moreover, FSP has given a clear indication about enhancement of microstructure in depth of the substrate. Also, the discrepancy in the value of micro hardness with escalating distance from the crest to the bottom has shown increased micro hardness on the top surface of the specimen. In the present study, it was observed that for the defined parameters the temperature produced during FSP was around 1173K . Reduction in grain size with an increase in strength of grain boundary entirely works according to Hall-Petch equation as discussed in section 3.2 and is considered to be the most important aspect that contributed towards increased hardness of the C70 steel[26]. Figure 10 depicts that grain size is inversely proportional to hardness conferring Hall- Petch.

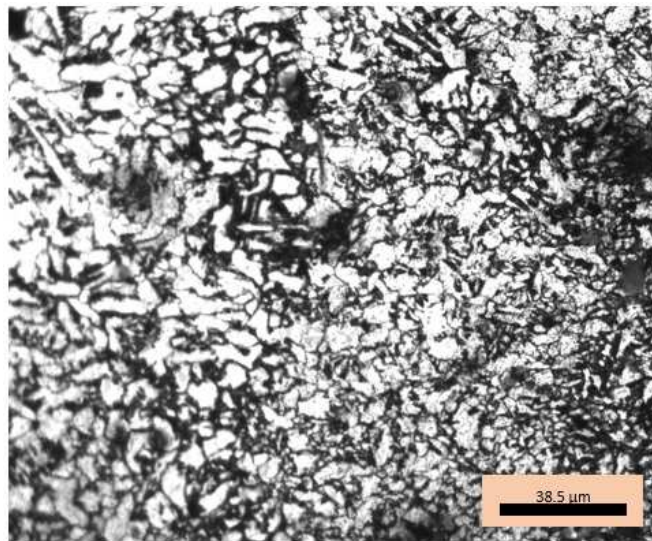


Figure 9: Microstructure of F Sped Sample Processed At 600 RPM

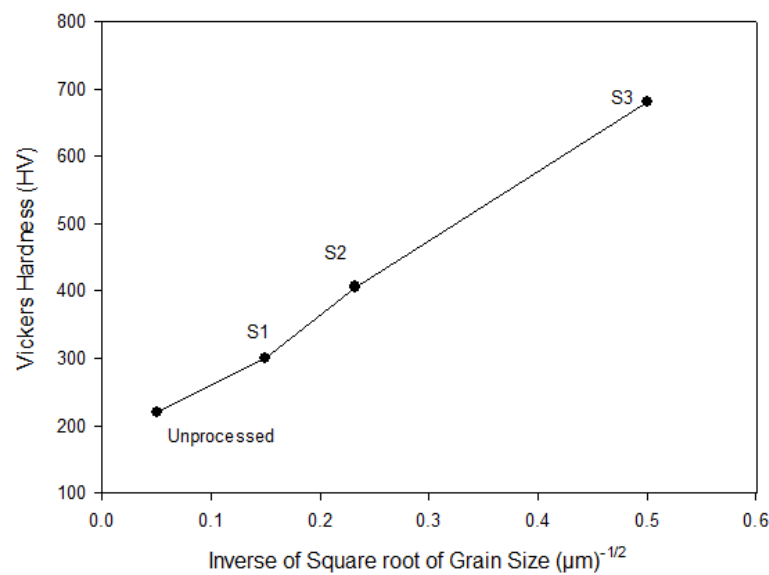


Figure 10: Plots Showing Relationship between Hardness and Grain Size According to Hall-Petch Equation

Scanning Electron Microscopy

SEM image of C70 steel friction stir processed at optimized parameters comprising 600 RPM single pass, 1mm plunge depth, 70mm min^{-1} feed rate is shown in Figure 11(b)

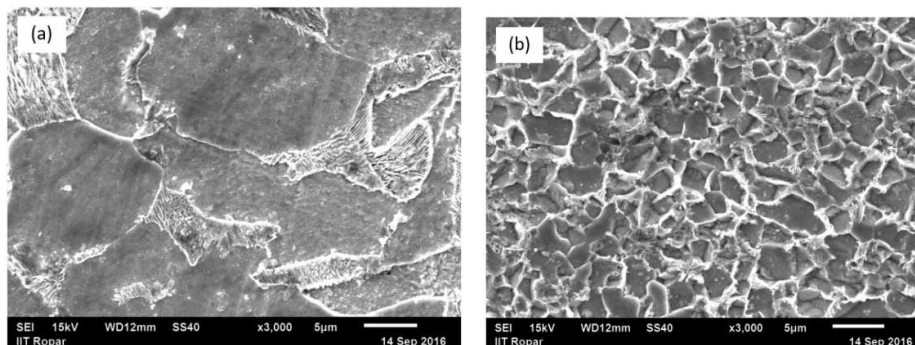


Figure 11: SEM Image of (a) as Received and (b) FSPed with Reduced Grain Size

The SEM images taken from the upper most part of the specimen undoubtedly depicts outwards orientation of precipitates. The microstructure refinement resulted in nearly three times enhancement of FSPed steels in comparison to BM as shown in Figure 11 (a). The SEM image shows the presence of grain size nearly $2-3\text{ }\mu\text{m}$ and extremely fine precipitates are also seen. The observed images of FSPed steel have shown no macroscopic defects such as cracks or porosity.

CONCLUSIONS

- High carbon steel was friction stir processed at room temperature to enhance its strength and refine the grain structure. The important conclusions of this investigation are summarized as follows: -
- C70 steel was successfully Friction stir processed in 150, 300, 600 RPM with 70 mm min^{-1} feed rate and 1 mm plunge depth by a W-C pin less tool of 8mm diameter.
- C70 steel was cooled by dry ice in an isolated system, resulting in phase transformation from ferrite to martensite.
- Some ultrafine dual phase structure of martensite and ferrites were characterized. An increase in the hardness and tensile strength was attained in the processed zone. Hardness value of FSPed steel increased from 220 HV to 681 HV with high UTS of 1190 MPa which is quite higher than 640MPa of BM. A subsequent decrease in the elongation to 7% resulted in reduced ductility.
- Microstructure refinement played a major role in reducing the grain size to an average of $2-3\text{ }\mu\text{m}$ after processing $20\text{ }\mu\text{m}$ grain size.
- A ductile fracture of a tensile specimen with big dimples was observed in case of BM whereas, FSPed samples exhibited a brittle fracture with small dimples.

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